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Effect of ion beam treatment (Ar^+, E= 30 keV) on the microstructure of titanium alloys

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Abstract. The effect of continuous Ar^+ ion beams (E = 30 keV, j = 400 μA/cm^2; F = $5{\cdot}10^{16}$ cm^{-2}) on the hardness and microstructure of the Gr2-titanium-based and Ti-6Al-4V alloys after cold working and subsequent recrystallization annealing is studied. It has been found that the initial microstructure of the annealed alloys in the equilibrium state, which consisted of recrystallized equiaxed grains, does not change after irradiation; thus the hardness of the irradiated samples is comparable with that of the initial samples. The initial fine fiber structure of the deformed Ti-6Al-4V alloy changes insignificantly after both annealing (T = 790°C, 30 min) and ion irradiation under used conditions. In contrast to that, the effect of rapid radiation annealing of the cold-worked titanium Gr2 alloy with beams of accelerated ions of inert gas at low temperatures (lower by 130°C than the conventional annealing temperature of these alloys at 680°C) and for a shorter time (9 min instead of 35 min, τ_{irr} ~ 20 s) has been revealed. The result is the formation of a uniform recrystallized structure with equiaxed fine grains 5-10 μm in size in the entire volume of the 3-mm-thick samples, despite the fact that the average projected range of 30-keV Ar^+ ions in titanium is only 20 nm.

1. Introduction

Examples of the successful use of accelerated ion beams in industrial processes to modify the electrical, magnetic, mechanical, contact-chemical, and other material properties have emerged in recent years [1-10]. A number of technological ion sources [2, 11, 12] capable of generating ion beams of various chemical elements with energies from a few to several tens of keV are developed and successfully used in manufacture.

However, the main problem of accelerated-ion-beam-based technologies used to modify the physical and mechanical properties of metals and alloys is a little depth of the impact zone. In normal conditions it is limited by a zone of the accelerated ion range, the length of which at ion energies obtained in the technological accelerators (<10^6 eV) does not exceed several tens or, in the best case, several hundred nanometers [13].

Authors of many published works dedicated to irradiation of metals and alloys with accelerated ion beams studied structural changes in the ion range zone and tried to find a way to increase the ion penetration depth using heating (i.e., thermally-stimulated processes) and increased ion energy. However, all these attempts to use high-energy ions (> 10 MeV) and powerful ion beams in order to increase the ion penetration depth deeper than several tens of micrometers, which is not sufficient for a great number of commercial applications, have failed.

The use of radiation-dynamic long-range effects [7-10] increases substantially the depth of the modified zone under ion-beam processing. These effects are caused by the generation of nanosized post-cascade shock waves (during evolution of dense cascades of atomic collisions) and propagation of these waves in a medium rather than by the doping of thin surface layers and the formation of radiation defects in them. These waves can initiate the plastic flow of the material on their front and phase transformations that are similar to combustion and detonation phenomena; because of this, changes in the structure and properties can be observed at the submillimeter and millimeter depths [8-10].

In particular, it was established in our works studying the influence that ion beams have on the structure and properties of various metastable media [10, 14] that under certain circumstances ion bombardment can be an alternative to furnace annealing. Radiation annealing in alloys occurs at significantly lower temperatures (by 150-200 K) and energy consumption, and this process takes less time, as compared with isothermal furnace annealing. The giant depth of the influence of accelerated ions (from 3 to 8 millimeters for commercial aluminum alloys [10, 14-16]) in the course of radiation annealing (at one-side irradiation) should be noticed. On the basis of these data, we obtained a patent for a method of treatment of commercial Al-Mg, Al-Cu-Mn-Mg, and Al-Li-Cu-Mg aluminum alloys with gas ion beams (Ar^+) with an energy of 40 keV, instead of prolonged (for 1-2 h) intermediate furnace annealing at increased temperatures (320-400 °C) [17].

It is of interest to study the possible application of radiation-dynamic effects under ion irradiation for the targeted modification of the structure and properties of other metals and structural materials based on them.

The aim of the work is to study the effect of Ar^+ ions with energy of 30 keV on the structure and properties of titanium-based Gr2 and Ti-6Al-4V alloys. In contrast to iron and aluminum, titanium as a basis for structural materials, owing to its small density (4.505 g/cm^3), large specific strength, incredible high corrosion resistance, and considerable strength at increased temperatures shows an advantage in aerospace engineering.

2. Experimental

The following alloys were used as the objects of the study: commercial titanium of Grade 2 (α alloy) and Ti-6Al-4V alloy (α+β martensite alloy). The titanium alloy sheets ~ 3 mm thick in different initial states were prepared at PAO "VSMPO-AVISMA Corporation" (Verkhnyaya Salda, Sverdlovsk region): after cold working and recrystallization annealing (Gr2: 680°C, 35 min; Ti-6Al-4V: 790°C, 30 min). The chemical compositions of the alloys are listed in Table 1. Unlike aluminum and its alloys, titanium has a high melting temperature and other peculiarities. For example, it has the ability to exist in two polymorphic states, namely, α titanium and β titanium, transitioning into one another at ∼ 882.5 °C [18]. Owing to the polymorphic α↔β transformation, a sufficiently large variety of structures can be obtained in titanium-based alloys. This, in turn, ensures a change in mechanical properties in a wide range.

The exposure to accelerated ion beams with an energy of 30 keV was carried out using an ILM-1 ion beam implanter equipped with a PULSAR-1M ion source based on a low-pressure glow discharge with a hollow cold cathode [11] in a continuous mode. A line-focus beam 20×100 mm^2 in cross section, under which plate-like samples of the alloys with dimensions of 90 × 210 × 3 mm^3 were moved back and forth at a speed of 1 cm/s, was cut out from Ar^+ ion beam of circular cross section using a collimator. The following beam parameters were specified: ion energy E = 30 keV, ion current density j = 400 μA/cm^2, and fluence $F = 5\cdot10^{16}$ cm^{-2}. The total time of treatment was approximately 9 min but the pure fluence accumulation time was about 20 s only. In the course of irradiation, the sample temperature was controlled with the help of a thin chromel-alumel thermocouple connected with an Advantech Adam 4000 automated system for digital signal registration. The maximum temperature of the samples was no higher than 550°C.

Brinell hardness and Rockwell hardness were measured using a TSh-2M and a TK-2M hardness testing machines, respectively, on both irradiated and nonirradiated sample sides. The metallographic analysis of the initial and irradiated samples was performed using a Neophot-30 optical microscope in the longitudinal and transverse directions over the entire cross section of a sample.

Table 1. Chemical composition of the studied titanium alloys.

Content of elements, wt. %							
Ti	Fe	O	H	N	C	Al	V
Gr2							
balance	0.3	0.25	0.015	0.03	0.1	-	-
Ti-6Al-4V							
balance	0.4	0.2	0.015	0.05	0.1	5.5-6.75	3.5-4.5

3. Results and discussion

The hardness of the samples of the titanium Gr2 and Ti-6Al-4V alloys in the various initial states and after irradiation with continuous Ar^+ ion beams are listed in Table 2.

The results listed in Table 2 suggest that the hardness of the *annealed* Gr2 and Ti-6Al-4V alloys remains the same after Ar^+ ion irradiation under used conditions. The resulting effect of ion irradiation on the deformed samples of the same alloys is different. Thus, irradiation decreases the hardness of the deformed Gr2 alloy to the values close to those of the annealed samples of this alloy. The hardness of the deformed Ti-6Al-4V alloy remains the same after irradiation.

Table 2. The hardness of the samples of the titanium Gr2 and Ti-6Al-4V alloys in the initial state and after irradiation with continuous Ar^+ ion beams.

Alloy	Alloy processing	HB		HRC	
		Side 1*	Side 2	Side 1	Side 2
Gr2	Cold working	244 ± 5	248 ± 5	26.5 ± 0.5	28.6 ± 0.5
	Cold working + irradiation (Ar^+, E = 30 keV, j = 400 μA/cm^2; $F = 5\cdot10^{16}$cm^{-2}; τ_{irr} ~ 20 s)	195 ± 5	189 ± 5	16.3 ± 0.5	15.3 ± 0.5
	Annealing at T = 680°C for 35 min air cooling	172 ± 5	167 ± 5	14.0 ± 0.5	12.0 ± 0.5
	Annealing + irradiation (Ar^+, E = 30 keV, j = 400 μA/cm^2, $F = 5\cdot10^{16}$ cm^{-2})	168 ± 5	167 ± 5	12.5 ± 0.5	13.5 ± 0.5
Ti-6Al-4V	Cold working	323 ± 5	318 ± 5	38.4 ± 0.5	38.1 ± 0.5
	Cold working + irradiation (Ar^+, E = 30 keV, j = 400 μA/cm^2; $F = 5\cdot10^{16}$cm^{-2})	330 ± 5	329 ± 5	38.6 ± 0.5	38.3 ± 0.5
	Annealing at T = 790°C for 30 min, air cooling	322 ± 5	328 ± 5	39.2 ± 0.5	39.7 ± 0.5
	Annealing + irradiation (Ar^+, E = 30 keV, j = 400 μA/cm^2, $F = 5\cdot10^{16}$ cm^{-2})	334 ± 5	334 ± 5	39.4 ± 0.5	39.5 ± 0.5
Note: *1 – irradiated side; 2 – nonirradiated side.					

Figure 2 demonstrates the microstructure of the Gr2 and Ti-6Al-4V alloys in various initial states. The cold-worked alloys exhibit a fine fiber structure (Figures 1a, 1c). The microstructure of the Gr2 alloy sheets 3 mm thick after annealing consists of recrystallized equiaxed grains, which are finer in the center over the transverse section than those along both surfaces. The grain size in the center of transverse section is ~ 25-40 μm (Figure 1b). In the Ti-6Al-4V alloy after annealing under used conditions (Table 2), a fine fiber structure retains (Figure 1d), but the fibrous structure becomes less pronounced.

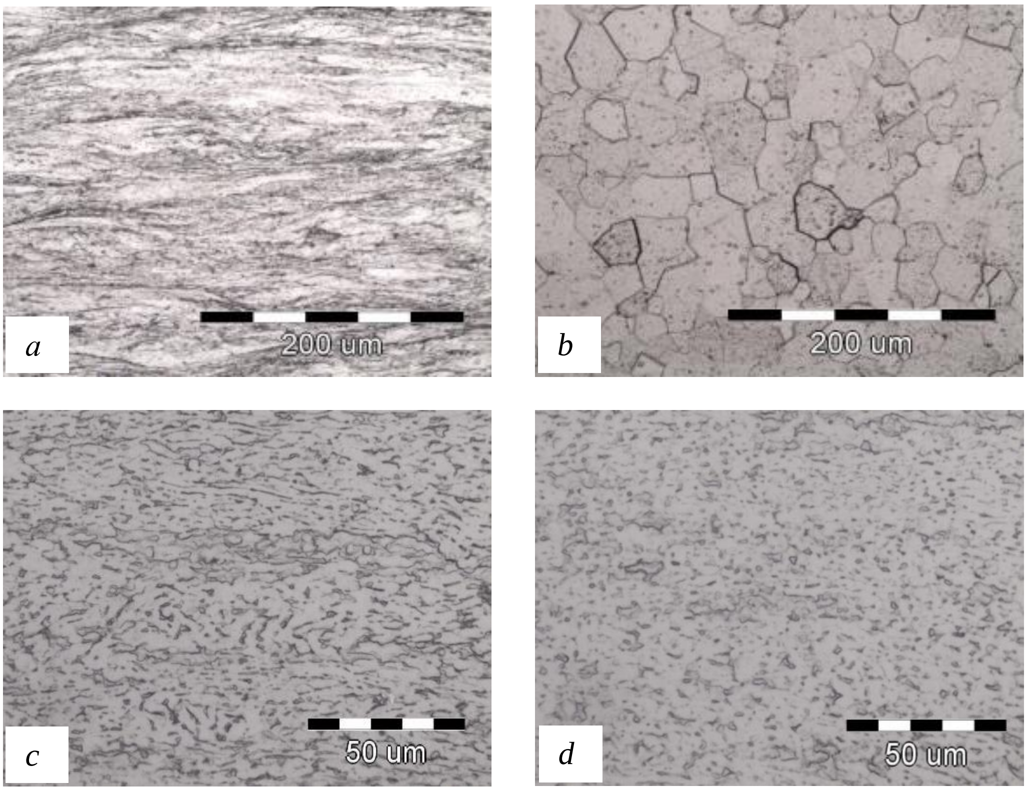

Figure 1. Microstructure of the sheets ~ 3 mm thick made of the (a, b) Gr2 and (c, d) Ti-6Al-4V alloys after (a, c) cold working and (b, d) recrystallization annealing; the central part of the samples over the cross section.

The irradiation of the deformed Gr2 alloy initiates recrystallization processes resulting in the formation of equiaxed fine grains 5-10 μm in size in the entire volume of the samples. Figure 1 demonstrates the microstructure of the Gr2 alloy over the cross section of sample 3 mm thick after ion irradiation. It can be seen that the irradiation-annealing-induced recrystallized structure is the same on the irradiated side, in the central part of the sample, and on the nonirradiated side. The average projected range of Ar^+ ions with an energy of 30 keV in titanium is only ~ 20 nm [13]. In this case, the processes observed over the large depth are caused by the radiation-dynamic effects (related to the propagation of nanosized post-cascade shock waves in medium) [10].

It should be noted that radiation-induced annealing of the deformed Gr2 alloy occurs during heating of the samples (without holding) to a temperature of 550°C. This is lower by 130°C than the conventional annealing temperature of these alloys (680°C). Furthermore, the irradiation annealing takes place for a shorter time (9 min instead of 35 min). Pure time of radiation annealing is ~20 s. The size of the recrystallized grains after ion beam treatment is smaller than that formed during furnace recrystallization annealing, which, as a rule, have a positive effect on the processing plasticity of the alloy.

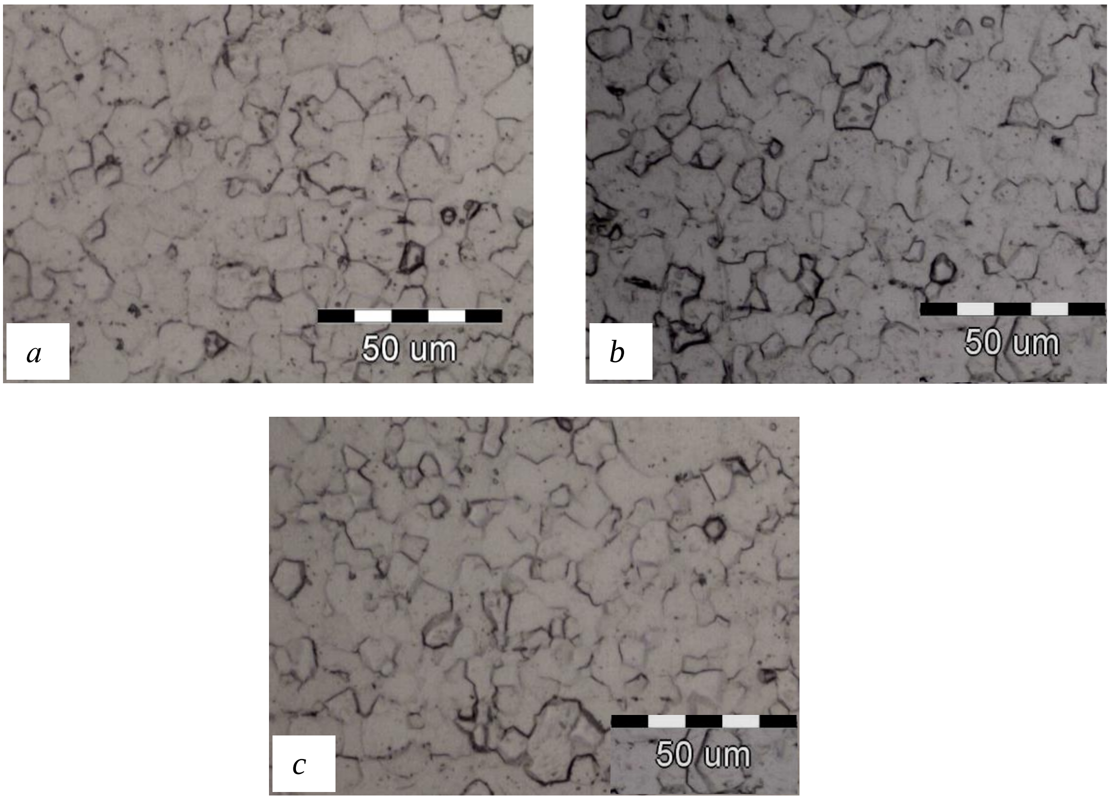

Figure 2. Microstructure of the Gr2 alloy after ion irradiation over the cross section of a sample 3 mm thick: (a) irradiated side, (b) central part of the sample thickness, and (c) nonirradiated side.

After irradiation of the deformed Ti-6Al-4V alloy, the initial fine fiber structure is retained. It is well known that alloyed titanium alloys, in particular, Ti-6Al-4V, at the same deformation degree have a higher recrystallization temperature than Gr2, since such alloying elements as Al and V markedly increase this temperature. It should be noted that the deformed microstructure has remained also after full recrystallization annealing at 790°C for 30 min (see Table 2). Two-phase titanium alloys are characterized by the fact that a marked change in the microstructure can be observed only during isothermal annealing [18], which includes alloy heating to relatively high temperatures of ~ 900-950°C, which are sufficient for recrystallization processes. Moreover, slow cooling to temperatures, which provide a high stability of the β phase and the final cooling in air to room temperature, are required.

Irradiation of the <u>*annealed*</u> Gr2 and Ti-6Al-4V samples under used conditions does not lead to the change in the microstructure. The samples are in an equilibrium state after annealing in contrast to the deformed metal, which possesses an increased internal energy and is thermodynamically unstable. Therefore <u>*radiation-dynamic*</u> effects (observed at low and very *low fluences*) do not show up.

Long-range effects <u>of the other nature</u> [4] are observed under *high-dose* irradiation (10^{17}-10^{18} cm^{-2}) of the annealed materials. They are related with the accumulation of high <u>*static*</u> stresses created by implantation of impurities (capable to exceed the yield strength of the material) and the formation of dislocation loops in the surface layer. These stresses ensure the movement of the generated dislocations deep into material (to few tens of μm). Such effect has not been observed in our case, since the fluence was relatively low.

4. Conclusion

It was established that the initial microstructure, which consisted of recrystallized equiaxed grains, was retained after Ar^+ ion irradiation of the *annealed* titanium Gr2 and Ti-6Al-4V alloys under used conditions (E = 30 keV, j = 400 μA/cm^2; $F = 5\cdot10^{16}$ cm^{-2}). The hardness of the irradiated samples was comparable with that of the initial samples. In the case of the irradiation of *cold-worked* Ti-6Al-4V alloy samples, the initial structure and the hardness remained the same. On the contrary, irradiation of the *cold-worked* Gr2 alloy samples 3 mm thick with the initial fine fiber structure reduced its hardness (to the same value on both the irradiated and nonirradiated sample sides). This was associated with the recrystallization in the entire volume of samples, resulting in the formation of fine equiaxed grains of 5–10 μm in size. The average projected range of 30-keV Ar^+ ions in titanium was ~ 20 nm only, which is more than 10^5 times greater than the depth of the impact.

Radiation-induced annealing of the deformed Gr2 alloy occurred during heating of the samples (without holding) to a temperature of 550°C. This is lower by 130°C than the conventional annealing temperature of these alloys (680°C). The treatment time decreased from 35 to 9 min (τ_{irr} ~ 20 s). Thus, this work shows the possibility of rapid radiation annealing of the titanium Gr2 alloy with beams of accelerated ions of inert gas at low temperatures at the expense of radiation-dynamic effects.

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